

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Application of:

MEADOR, et al.

Serial No. : 10/784,045

Filed: February 20, 2004

THERMALLY CURABLE MIDDLE LAYER  
FOR 193-NM TRILAYER RESIST PROCESS

Docket No.: 32849

Confirmation No.: 6833

Group Art Unit No.: 1712

Examiner: Moore, Margaret G.

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Commissioner for Patents  
P.O. Box 1450  
Alexandria, VA 22313-1450

Sir:

DECLARATION UNDER 37 C.F.R. § 1.131

1. I, Jim D. Meador, am one of the inventors named in the above-referenced patent application. I am making this declaration to establish invention of the subject matter claimed in the above-referenced patent application on a date prior to August 5, 2003, the filing date of U.S. Patent Publication No. 2005/0031964 by Babich et al.

2. Pages 43-44 from lab notebook 2404 are attached as Exhibit A. Exhibit A describes a composition within the scope of independent composition claims 41, 89, and 95. Each limitation of these claims can be found in Exhibit A. Page 43 of Exhibit A describes the preparation of a polyhedral oligomeric silsesquioxane ("POSS") with an alcohol functionality as claimed. That is, methacryl ethyl-POSS is polymerized with hydroxypropyl methacrylate to form a POSS polymer having an alcohol (-OH) functionality.

3. Page 44 of Exhibit A describes using the POSS having an alcohol functionality (discussed in Paragraph 2 above and referenced as "JM2404-43") to prepare a composition. The POSS having an alcohol functionality is dissolved or dispersed in a solvent system (PGMEA in this instance). As recited in claims 41 and 95, the composition includes a cross-linking agent (i.e., PWL, which is an abbreviation for the aminoplast crosslinking agent sold under the name Powderlink® 1174). Claim 89 also recites the presence of a strong acid and a weak acid. This is also shown on page 44 of Exhibit A in that the composition includes both *p*-TSA (*p*-toluenesulfonic acid monohydrate, a strong acid that is also a catalyst) and bisphenol S (a weak acid). Page 7, lines 8-18 of the application as filed define strong and weak acids and list examples of each. Bisphenol S is listed as a preferred weak acid, while *p*-TSA is listed as a preferred strong acid.

4. The next-to-last paragraph on page 44 of Exhibit A describes how the composition was spin-coated onto a substrate at 1,500 rpm followed by baking to give a layer having a thickness of 669.7 Å. The baked film was insoluble in ethyl lactate. These steps, in combination with the use of the composition described in Paragraph 3 above, shows the presence of each of the limitations of independent structure claims 53 and 91 and independent method claims 71 and 94.

5. The dates on Exhibit A have been blocked out. However, I declare that these dates are earlier than August 5, 2003, and that they show invention by my coinventors and me of the claimed subject matter prior to August 5, 2003.

6. A page entitled "HML JM2404-44 on ARC29A with GARS8107A10: Processing Conditions" is attached as Exhibit B. "JM2404-44" refers to a composition described in Paragraph 3 above. Exhibit B shows the stack and processing conditions (spin speeds, curing conditions, film thicknesses, exposure and developments steps) used to form a layer of the composition of Exhibit A on a substrate coated with a bottom anti-reflective coating, to apply a photoresist (GARS8107A10) to that composition layer, and to pattern the photoresist. Thus, the photoresist limitation recited by independent claims 90 and 92-93 is provided by Exhibit B. Of course, the remaining limitations of claims 90 and 92-93 are described earlier in this Declaration.

7. The date on Exhibit B has been blocked out. However, I declare that these dates are earlier than August 5, 2003, and that they show invention by my coinventors and me of the claimed subject matter prior to August 5, 2003.

8. One of our initial goals behind working with the POSS materials was the desire to maximize the amount of silicon in the compositions. The idea of high silicon content has generic applicability to the use of silicon-rich POSS materials in the compositions, whether the POSS is included in the composition directly or as a POSS compound, or is present as a co-monomer in a polymer.

9. The composition prepared in Exhibit A involved the polymerization of a POSS compound to form a POSS polymer having an alcohol functionality. The composition comprised

of this POSS polymer demonstrated that POSS materials did have high silicon content and were amenable to middle or protective layers for lithographic processes. After the POSS polymer, I went directly to trying a POSS compound, octahydroxypropyldimethylsilyl-POSS. Page 53 of notebook 2404 is attached as Exhibit C and shows work with the octahydroxypropyldimethylsilyl-POSS. I did not try any other POSS polymers prior to trying the octahydroxypropyldimethylsilyl-POSS compound.

10. I further declare that all statements made herein of my own knowledge are true and that statements made herein on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application, any patent issuing thereon, or any patent to which this Declaration is directed.

Jim D. Meador  
Jim D. Meador

August 20, 2007  
Date

PROJECT

Copolymerization Methyl Methacrylate - POSS and Hydroxypropyl Methacrylate

Continued From Page

MA0717, Methyl Methacrylate, P-POSS is soluble in PGMEA at ambient conditions

A 100 ml three-necked flask equipped with magnetic stirring bar, thermometer, nitrogen inlet, and condenser with nitrogen outlet was charged with 4.00g (5.35 mmol) of methyl methacrylate (MA0717), 2.67g (20.8 mmol) of hydroxypropyl methacrylate (268542-1L), 60.7g (60.7 mmol) of AIBN (44,109-0). The mixture was flushed with nitrogen at ambient conditions from 11:13 AM Wed. to 11:28 AM Wed. and the flask then remained in an oil bath at 65°C. The mixture was stirred under nitrogen. Soln. temp 60°C at 11:45 AM Wednesday. Soln. temp 62°C at 11:57 AM Wed. Soln. temp 62°C at 12:10 PM Wednesday. Soln. temp 61°C at 1:00 PM Wed. Soln. temp 61.5°C at 1:24 PM Wed. Soln. temp 63°C at 3:32 PM Wed. Soln. temp 61.5°C at 4:55 PM Wed. Soln. temp 61°C at 8:07 AM Thursday. Soln. temp 62°C at 11:10 AM Thursday. Soln. temp 60.5°C at 11:55 AM. Then and with the nitrogen still flowing the flask was removed from the oil bath. With the solution temperature at 26.5°C, 17.1 mg of 4-methoxyphenol (M1, 845-5) was added. The mixture was stirred from 12:34 PM to 1:04 PM = 30 min. On adding a drop of the solution to methyl alcohol (M1239), a precipitate was not observed which is CHLOROS. Quantitative yields of product = 66.55g. There was a very small amount of insoluble in the solution. Upon adding a drop of the M-L to ethanol (179410) still observed no precipitate.

Order of Charge Recipe

3	MA0717	4.00g	[120g of silicon]
1	hydroxypropyl methacrylate	2.67g	
2	PGMEA	60.7g	
4	AIBN	66.6mg	55.2mg indistinguishable

67.43g Theoretical Yield

Percent silicon in the polymer =  $1.20 / 6.725 \times 100 = 17.94$

After adding the 4-methoxyphenol theoretical yield

9.97% polymer solids in 67.45g. ∴ 0.2746 mmol hydroxypropyl.

24 hr at 60.5 - 63°C

Read and Understood By

Jan D. Meador

Signed

Date

Signed

Date

The polymer sol. M<sub>w</sub> was 55,700  
M<sub>n</sub> was 20,200  
D was 2.76

Continued on Page

Preparation of an EML from  
JM2404-43

Notebook No. 2404

Continued From Page

A 250 ml Nalgene bottle was charged with the following ingredients in the stated order and the mixture stirred to homogeneity at ambient conditions.

(M-L) JM2404-43 30.0g [2.99% polymer, 8.238 mg hydroxyl, 27% PGMEA]  
PGMEA 89.8g  
PWL (LI 3/24/03) 0.886g [8.24 mg]  $\text{H}_2\text{O} \text{ in PWL} = \frac{0.886}{0.886 + 2.78} \times 100 = 24.86\%$   
A-TSA-HO (40, 288-5) 36.5g  
bisphenol S [10, 303-4] 110.8g  
120.83g [3.33%]

The homogeneous solution was taken off the magnetic stirring plate on Friday afternoon. Rather than decanting the solution was taken directly to the clean room. A spin at 1500 rpm for 60 sec, followed by a cure at 205°C / 60 sec - we gave 669.7A. A small amount of smoke was given off during the bake. Ethyl lactate strip was +0.59%. Polymerization did occur!

About 11:15, A the EML was damaged by tumbling with 5.8g of PGME-washed 650C beads from 11:31 AM Monday to 3:27 PM Monday (4hr). The beads were removed by straining thru 2 layers of plastic cloth. The EML was then twice filtered thru a 0.1um int-port into 125 ml Nalgene bottles.

Continued on Page

Read and Understood By

Ken D. Meador

HML JM2404-44 on ARC29A with GARS8107A10:  
Processing Conditions

-2-

ARC29A-8

Bake: 205 °C/60 sec.  
Thickness: 77 nm

EML JM2404-44

Coating: 1250 rpm/60 sec.  
Bake: 205 °C/60 sec.  
Thickness: 73.6 nm \*

\* BSI data

Resist: GARS8107A10  
Modified acrylate, dense lines  
Dispense: Manual  
SB: 115°C/90 sec.  
Thickness 200 nm  
Exposure Tool:  
ASML 5500/1100,  
NA 0.75, \_ 0.89/0.55  
Dipole illumination  
Mask: TM99LF 9%attPSM  
E 16.0+0.5 mJ/cm<sup>2</sup>  
F 0.0+0.1  
FEM E15xF15  
PEB: 110°C/90 sec.  
Develop.:OPD262-LD60 sec.



Mariya Nagatkina

Reference to IMEC is obligatory for representations



© imec

tabbles

EXHIBIT

B

This Hybrid Plastic monomer is soluble in PGMEA at ambient conditions.

A 125 ml Nalgene was charged with 1.5265g (8.24 mmol) hydroxypropyl <sup>cf ALO35</sup> 61.3g of PGMEA, 0.66g (6.14 mmol) of PNL 1174, 27.2 mg of p-TSA, 16.0 <sup>(10,188)</sup> and 82.3 mg of bisphenol S [10,303-9]. The mixture was stirred at ambient conditions overnight.

ALO35 = octahydroxypropyl dimethylallyl P-POSS.

Stirring continued until 1:00 PM Tuesday, at which point we had a solution.

About 3.5g of PGME-washed 650C beads were added and the mixture tumbled from 1:17 PM Tuesday to 5:18 PM Tuesday. The beads were removed by straining thru two layers of plastic cloth.

Additional processing was by Denise!

This will include filtration.

The dried film, after removing the solvent, will contain 20.5% silicon.

Continued on Page

Read and Understood By

Jen D. Meador

Signed

Date

Signed

Date

tabbles

EXHIBIT